

Docket 86430AJA  
Customer No. 01333

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**  
**BEFORE THE BOARD OF PATENT APPEALS AND INTERFERENCES**

In re Application of:  
Rajesh V. Mehta, et al

PROCESS FOR THE FORMATION  
OF PARTICULATE MATERIAL

Serial No. US 10/814,354

Filed 31 March 2004

Group Art Unit: 1723

Examiner: Joseph W. Drodge

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Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

Sir:

**APPEAL BRIEF PURSUANT TO 37 C.F.R. 41.37**

Applicants hereby appeal to the Board of Patent Appeals and  
Interferences from the Examiner's Final Rejection of claims 1-8 and 10-18 which  
was contained in the Office Action mailed July 19, 2007.

A timely Notice of Appeal was electronically filed October 19, 2007.

Respectfully submitted,

  
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### **Real Party In Interest**

Eastman Kodak Company is assignee and the real party in interest.

### **Related Appeals And Interferences**

No appeals or interferences are known which will directly affect or be directly affected by or have bearing on the Board's decision in the pending appeal.

### **Status Of The Claims**

Claims 1-8 and 10-18 are pending in the application.

Claim 9 has been cancelled.

Claims 1-8 and 10-18 stand rejected under 35 USC § 103.

Claims 1-8 and 10-18 are being appealed.

Appendix I provides a clean, double spaced copy of the claims on appeal.

### **Status Of Amendments**

No amendment has been filed after the Final Rejection dated July 19, 2007.

### **Summary Of Claimed Subject Matter**

Independent claim 1 is directed towards a process (page 6, lines 5-30) for the formation of particulate material (page 8, lines 3-24) of a desired substance comprising: (i) charging a particle formation vessel (10, new Fig. 9 filed June 8, 2007), the temperature and pressure in which are controlled (page 8, line 29 to page 9, line 3; page 17, lines 24-26; page 18, lines 13-15; page 19, lines 8-10; page 20, lines 2-4; page 20, line 32 to page 21, line 1), with a supercritical fluid (page 9, lines 3-4); (ii) agitating the contents of the particle formation vessel with a rotary agitator (11, new Fig. 9; page 9, line 5 to page 12, line 26) comprising an impeller (12, new Fig. 9) having an impeller surface (s, new Fig. 9) and an impeller diameter (d, new Fig. 9), creating a relatively highly agitated turbulent flow zone (13, new Fig. 9; page 7, lines 30-31) located within a distance of one impeller diameter from the surface of the

impeller of the rotary agitator, and a bulk mixing zone (14, new Fig. 9) located at distances greater than one impeller diameter from the surface of the impeller; (iii) introducing into the agitated particle formation vessel at least a first feed stream comprising at least a solvent (page 8, lines 25-29) and the desired substance dissolved therein through a first feed stream introduction port (15, new Fig. 9) and a second feed stream comprising the supercritical fluid through a second feed stream introduction port (16, new Fig. 9), wherein the desired substance is relatively insoluble in the supercritical fluid relative to its solubility in the solvent and the solvent is soluble in the supercritical fluid (page 8, line 29 to page 9, line 3), and wherein the first and second feed stream introduction ports are located within a distance of one impeller diameter from the surface of the impeller of the rotary agitator such that the first and second feed streams are introduced into the highly agitated zone of the particle formation vessel and the first feed stream is dispersed in the supercritical fluid by action of the rotary agitator (page 9, lines 5-9), allowing extraction of the solvent into the supercritical fluid (page 9, lines 1-3), and (iv) precipitating particles of the desired substance in the particle formation vessel with a volume-weighted average diameter (page 9, lines 14-16) of less than 20 nanometers (Example 6, page 19, lines 15-16; as well as Example 4, page 17, line 31; Example 5, page 18, lines 19-20; Example 7, page 20, lines 8-9; and Example 8, page 21, lines 6-7).

### **Grounds Of Rejection To Be Reviewed On Appeal**

1. Claims 1-8 and 10-18 are rejected under 35 U.S.C. 103(a) as being unpatentable over Saim et al (US 6,858,166) in view of Johnson et al (US 2004/0091546) and O'Conner et al (US 2006/0124783).

### **Arguments**

#### ***Obviousness Rejection of Claims 1-8 and 10-18 over Saim et al in view of Johnson et al and O'Conner et al***

The Examiner states that Saim et al ("Saim") disclose formation of micro or nano-particles by a process of admitting a supercritical fluid to a vessel, in which temperature and pressure are controlled (column 14, lines 21-45), agitating such vessel with a rotary agitator comprising an impeller of un-specified, given diameter relative to

vessel diameter (column 14, line 63 column 15, line 6), introducing a 1<sup>st</sup> feed stream comprising a solvent and desired, active substance through a 1<sup>st</sup> introduction port and introducing a 2<sup>nd</sup> feed stream comprising the supercritical fluid through introduction ports both approximately within the highly agitated zone of the mixer that may be an impeller (see especially figures 1 and 2 and column 18, lines 30-63). The Examiner further states that both a first feed stream including particle-forming components and solvent and a second feed stream containing supercritical fluid may be introduced proximate the agitated/highly agitated zone of the mixer (see column 12, lines 11-12 taken with lines 33-36 of column 12), and that particles are then precipitated within such vessel over a carrier bed.

The Examiner further states that O'Connor et al ("O'Connor") teaches to produce nanoparticles using solvents and supercritical fluids by use of conversion/mixing vessels that combine impeller mixers with other type stirrers, that have inlets for introducing solvents and other materials, and/or have a plurality of impeller mixers or impellers with differently functioning blades so as to create different mixing zones of different degrees of turbulence, and that it would have been obvious to one of ordinary skill in the art to have adapted the more-complex configuration of mixing/agitating means of O'Connor in the process of Saim, to effect greater, more complete mixing of components which are in slurry form, or mixing of materials of different phases (liquids, solids, semi-solids and gases). The Examiner further states Johnson et al ("Johnson") teach production of nanoparticles using supercritical fluid processing in which the inlet tubes are within 15% of the agitator surface diameter (see especially paragraph 44, paragraphs 39-42, 58 and 63), and that it would have been obvious to one of ordinary skill in the art to have located the end of the inlet tubes of Saim very close to the impeller agitators as suggested by Johnson, to facilitate rapid incorporation of the incoming fluid into the swept region of the agitator and rapid mixing.

This rejection represents clear error, as the Examiner has misinterpreted the individual teachings of the cited references, and has further failed to establish a prima facie case of obviousness with regard to combining the teachings of such references so as to arrive at the present claimed invention, as each of such references employ distinct types or materials and or apparatuses for distinct purposes, and as the proposed combinations and modifications of the individual references go against the expressed preferences of the individual references.

Saim, e.g., is directed towards trapping (e.g., coating or dispersing) of precipitated particles in a bed of powder carrier material. While Saim teaches that the bed may be agitated to uniformly distribute precipitated solute particles throughout the mixed powder bed (col. 10, lines 59-67), such mixing is not even required, and such trapping or coating is not necessarily a rapid kinetic process as compared to the micromixing time scales associated with the precipitation of particles themselves. While a rotary agitator is disclosed for agitation of the bed of carrier material, Saim does not teach that both the first feed stream including particle-forming components and solvent and the second feed stream containing supercritical fluid should be introduced proximate an agitated/highly agitated zone of the mixer for the purpose of formation of discrete particles having a size of less than 20 nm. In such connection, the Examiner's reliance upon column 12, lines 11-12 taken with lines 33-36 of column 12 is clearly in error, as lines 11-12 are directed towards a first "Mode 1" employing RESS (Rapid Expansion of Supercritical Solutions) techniques (and thus there are not separate first and second streams introduced into the vessel, but rather only a single pressurized stream which is expanded upon entry into the vessel), while lines 33-36, which are directed towards a SAS (Supercritical Anti-Solvent) type process more similar to the present process, simply do not teach entry of the separate solutions and pressurized gaseous fluid in a highly agitated zone within one impeller diameter of the agitator impeller surface. Similarly, the Examiner's further reference in the Final Rejection to column 12, lines 8-36, column 13, lines 14-30, and column 15, lines 15-22 of Saim with respect to introduction of feed streams also does not teach such required feature of the claimed invention. To the contrary, Saim discloses in such cited sections a preference for the introduction of the pressurized gaseous fluid only from above an upper surface of the bed of carrier particles (i.e., clearly away from any highly turbulent zone that may be created by the mixer), and of the organic liquid solution from a level below or slightly above the upper surface of the bed of carrier particles, and mixing of the bed of carrier particles to coat the carrier particles with particles of material precipitated from the solution.

Accordingly, whether the impeller depicted in Fig 2 of Saim inherently creates two mixing zones or not as argued by the Examiner, there is simply no teaching or suggestion to introduce both feed streams into any created highly agitated zone created within one impeller diameter of the impeller surface so as to enable formation of discrete particles having a size of less than 20 nm, especially in view of the explicitly stated

preferences for other introduction point locations taught by Saim discussed above. As Saim is clearly directed toward obtaining a specific result different from that of O'Connor and Johnson (i.e., coating of particles in a fluidized particulate bed), it clearly would not be "obvious" to change such process away from the specifically described preferences based on references which are not directed towards achieving such desired result of Saim.

It is further noted that the O'Connor process, although employing impellers and supercritical fluids, is directed towards the size reduction of pre-made macro particles, rather than the direct precipitation of small sized particles as is Saim. The physics of size reduction is fundamentally different from that of particle formation via precipitation, and accordingly no reasonable extrapolation can be made from the proposed combination of the Saim process with the teachings of O'Connor. The Examiner has provided no reasonable explanation as to why one skilled in the art would combine the particle reduction process of O'Connor with the particle precipitation process of Saim, especially to the extent such combined teachings would be inconsistent with the expressly taught preferences of the process of Saim.

Regarding Johnson, the referenced teaching in paragraph [0044] thereof of employing inlet tubes which are within 15% of an agitator surface diameter is essentially duplicative of the already acknowledged prior art mixing technology discussed at page 9, lines 24-31 and the paragraph bridging pages 10-11 of the specification. What is not taught or suggested, by either Johnson or the acknowledged prior art mixing technology, is to employ such type known type of mixing technology apparatus in a SAS type particle formation process. To the contrary, Johnson is directed towards making of nanoparticles of amphiphilic copolymers in conventional liquid solvents. In such connection, referenced paragraph [0058] of Johnson is not directed towards a teaching of supercritical fluid introduction during the process of nanoparticle formation as alleged by the Examiner, but rather discloses the use of liquefied gas (not a supercritical fluid) as a non-process solvent, while referenced paragraph [0063] states that the final solvent containing the formed amphiphilic copolymer nanoparticles can be subsequently altered by a supercritical fluid extraction post treatment process. Accordingly, there is no support for the Examiner's assertion that Johnson teach production of nanoparticles using supercritical fluid processing, and the process of Johnson does not enable production of particles having a volume-weighted average diameter of below 20 nm as required by the present claimed invention.

It is further noted that Johnson actually teaches that an agitator is not even required in the disclosed process if the fluids added into a non-solvent have a high mixing velocity sufficient to mix the fluid contents rapidly and in a controlled fashion (see, e.g., last 4 lines of paragraph [0017]). Supercritical fluids as required in the present invention are known to have gas-like transport properties, as noted at page 1, line 10 of the specification. For micromixing, the critical transport property of interest is molecular diffusivity. Gas-like molecular diffusivity lowers mixing time to levels not attainable in liquids. Thus, Johnson's teaching that an agitator is not needed where the fluids are combined in a manner otherwise providing a high mixing velocity in fact would appear to teach that a mixer would not be required for mixing in supercritical fluids (as the gas-like transport properties of super-critical fluids itself provides high molecular diffusivity), thus teaching away from the present invention, which is consistent with the preferences taught in Saim of adding the supercritical fluid above the mixed bed.

Finally, while it is believed that a *prima facie* case of obviousness with respect to the present claimed invention clearly has not been established based on the arguments above, it is further noted that any such alleged *prima facie* case of obvious is in any event overcome by the surprising results of the present invention. As disclosed and demonstrated in Examples 4-8 of the present application, the invention has been shown to enable production of particles of mean diameter less than 20 nm for a number of materials, many of them substantially smaller than even 10 nm, while none of the cited references teach a process which enables such small particle size. The Examiner's reference to column 9, lines 39-42 of Saim as a teaching of obtaining particles having diameters as small as 0.001 micron (1 nanometer) may be formed does not equate to an enablement of such mean particle size. Rather, Saim only uses such 0.001 micron as a lower limit for a definition of "nanoparticles". Saim has not demonstrated formation of discrete nanoparticles in the size range of less than 20 nm, and to the contrary, the designation therein at col. 9 lines 39-42 of nanoparticles "preferably" having an average particle diameter in the range of about 0.05 to 0.5 micron (i.e., 50 to 500 nm) is evidence of the non-obviousness of the present invention's ability to achieve particles have an average size of less than 20 nm.

**Conclusion**

For the above reasons, Appellants respectfully request that the Board of Patent Appeals and Interferences reverse the rejection by the Examiner and mandate the allowance of Claims 1-8 and 10-18.

Respectfully submitted,



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## Appendix I - Claims on Appeal

1. A process for the formation of particulate material of a desired substance comprising:
  - (i) charging a particle formation vessel, the temperature and pressure in which are controlled, with a supercritical fluid;
  - (ii) agitating the contents of the particle formation vessel with a rotary agitator comprising an impeller having an impeller surface and an impeller diameter, creating a relatively highly agitated turbulent flow zone located within a distance of one impeller diameter from the surface of the impeller of the rotary agitator, and a bulk mixing zone located at distances greater than one impeller diameter from the surface of the impeller;
  - (iii) introducing into the agitated particle formation vessel at least a first feed stream comprising at least a solvent and the desired substance dissolved therein through a first feed stream introduction port and a second feed stream comprising the supercritical fluid through a second feed stream introduction port, wherein the desired substance is relatively insoluble in the supercritical fluid relative to its solubility in the solvent and the solvent is soluble in the supercritical fluid, and wherein the first and second feed stream introduction ports are located within a distance of one impeller diameter from the surface of the impeller of the rotary agitator such that the first and second feed streams are introduced into the highly agitated zone of the particle formation vessel and the first feed stream is dispersed in the supercritical fluid by action of the rotary agitator, allowing extraction of the solvent into the supercritical fluid, and

(iv) precipitating particles of the desired substance in the particle formation vessel with a volume-weighted average diameter of less than 20 nanometers.

2. A process according to claim 1, further comprising (v) exhausting supercritical fluid, solvent and the desired substance from the particle formation vessel at a rate substantially equal to the rate of addition of such components to the vessel in step (iii) while maintaining temperature and pressure in the vessel at a desired constant level, such that formation of particulate material occurs under essentially steady-state continuous conditions.

3. A process according to claim 2, wherein the supercritical fluid, solvent and desired substance are exhausted from the particle formation vessel by passage to an expansion chamber.

4. A process according to claim 3, wherein the supercritical fluid, solvent and desired substance are exhausted from the particle formation vessel by passage through a backpressure regulator.

5. A process according to claim 3, wherein the supercritical fluid, solvent and desired substance are exhausted from the particle formation vessel by passage through a capillary.

6. A process according to claim 3, wherein the supercritical fluid, solvent and desired substance are exhausted from the particle formation vessel by passage through a flow distributor.

7. A process according to claim 3, further comprising collecting particles of the desired substance in the expansion chamber.

8. A process according to claim 1, wherein the supercritical fluid, solvent and desired substance are exhausted from the particle formation vessel directly into a solution to form a dispersion of the formed particles of the desired substance.

10. A process according to claim 1, wherein particles of the desired substance are precipitating in the particle formation vessel with a volume-weighted average diameter of less than 10 nanometers.

11. A process according to claim 10, wherein the coefficient of variation of the particle size distribution of the particles of the desired substance precipitated in the particle formation vessel is less than 50%.

12. A process according to claim 11, wherein the coefficient of variation of the particle size distribution of the particles of the desired substance precipitated in the particle formation vessel is less than 20%.

13. A process according to claim 1, wherein the coefficient of variation of the particle size distribution of the particles of the desired substance precipitated in the particle formation vessel is less than 50%.

14. A process according to claim 13, wherein the coefficient of variation of the particle size distribution of the particles of the desired substance precipitated in the particle formation vessel is less than 20%.

15. A process according to claim 1, wherein the desired substance comprises a colorant.

16. A process according to claim 15, wherein the desired substance comprises a dye.

17. A process according to claim 1, wherein the desired substance comprises a pharmaceutically useful compound.

18. A process according to claim 1, wherein the desired substance comprises a compound used to make organic electroluminescent devices.

**Appendix II - Evidence**

**NONE**

**Appendix III – Related Proceedings**

NONE